EXPLORATORY INVESTIGATION ON LOW-PRESSURE ROTATING DYNAMIC MEMBRANE PROCESS FOR TREATING BITUMEN/WATER/MINERAL EMULSIONS

FINAL REPORT

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FOR

CANMET, ENERGY, MINES AND RESOURCES CANADA

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SUMMARY AND CONCLUSIONS

Canada possesses large deposits of heavy oil/bitumen. The most commonly used technique for in-situ recovery of heavy oil/bitumen involves steam stimulation. The associated water requirements are quite high (the water-to-oil ratio may vary from 2:1 and up to 10:1). Therefore, an economically efficient recycling of "produced water" (i.e., condensed steam and formation water) is a recognized problem in the industry.

After separation of free oil, the produced water usually contains emulsified oil, suspended solids, dissolved organic and inorganic substances. The oil/water/mineral emulsion associated with in-situ production of heavy oil/bitumen are characterised by high stability. The use of conventional de-emulsifying processes proved to be ineffective.

Numerous methods and processes are being investigated for produced water treatment. Most of them have been adopted from classical boiler feedwater treatment principles. Among other processes being investigated is membrane technology. The recent investigation carried out by CANMET has shown the principal possibility to use prefabricated porous membranes for production of boiler-feed quality water from the wellhead bitumen/water/mineral emulsion containing about 4% bitumen. The subsequent experimental investigation carried out by Zenon Environmental Inc., on a contract to CANMET, has further confirmed the possibility of produced water treatment by using several types of prefabricated porous membranes.

The low-pressure rotating dynamic membrane (LPRDM) process is a variety of membrane technology that may be applied for separation of fine particles or substances of different molecular weight or specific gravity or both, from suspensions, emulsions, etc. Contrary to prefabricated porous membranes, the dynamic membranes are formed in-situ by a colloidal suspension

or solution flowing (circulating) through a porous support and thus depositing a thin micron-size layer which acts as a filter membrane. The porous structure, permeability and performance of the dynamic membranes can be adjusted by varying the membrane formation conditions (e.g., type of membrane forming solution, pH, concentration).

The low-pressure rotating dynamic membrane process (LPRDM) is a combination of an axial filter and a dynamic membrane formed and operated on a rotating porous surface of the filter.

The research and design work on an LPRDM unit and process was initiated by Dr. J.A. Mikhlin in 1977. Since that time, the mechanism of rotating dynamic membrane formation, as well as the performance of several types of these membranes, with both model compounds and different industrial effluents, suspensions and waste waters, have been investigated in the LPRDM process.

The current investigation on the LPRDM process for treatment of bitumen/water/mineral emulsions has been carried out by the Technical Development Department, SNC Inc., for CANMET.

The main objective of the current investigation was to explore the feasibility and effectiveness of treating a bitumen/water/mineral emulsion from a Canadian in-situ heavy oil/bitumen recovery operation using the LPRDM process.

A sample of bitumen/water/mineral emulsion from the in-situ recovery operation of Texaco Canada Resources, Fort McMurray, Alberta was used. The investigation was carried out with the emulsion sample, as received

and at increased concentrations of suspended and dissolved substances corresponding to reduction of initial emulsion volume by 50%, 90% and above 90%. The samples of increased concentrations were obtained from as received emulsion by ultrafiltration (separation of water) using the LPRDM process.

Two types of dynamic membranes, zirconium hydrous oxide (further designated as "Zr") and poly-2 vinylpyridine (PVP) have been used in most of the experiments. Also, in some tests, polyacrylic acid (PAA) dynamic membranes were formed.

The results obtained by Zenon Environmental Inc., using prefabricated ultrafiltration membranes for bitumen/water/ emulsion treatment were compared with those obtained in the current study. For comparison, the same emulsion sample was used in both SNC and Zenon Environmental investigations.

A summary of the results obtained during this exploratory investigation is presented below:

- Ultrafiltration of emulsions investigated in the LPRDM process gave positive results with all three types of dynamic membranes; Zr, PVP and PAA.

- A high degree of water recovery corresponding to reduction of initial emulsion volume by 90% and above was achieved.

- The quality of permeate (i.e., separated water) and rejection of various components by the dynamic membranes were comparable with those obtained using prefabricated ultrafiltration membranes.

SUMMARY AND CONCLUSIONS (cont'd)

- The average permeate flux in the LPRDM unit is 4- to 8-fold higher than that obtained with prefabricated ultrafiltration membranes, despite 10-fold lower transmembrane pressure (10 psi in the LPRDM unit versus 100 psi with prefabricated UF membranes).

- The performance characteristics of dynamic membranes during ultrafiltration of investigated emulsions (permeate flux and rejection of oil and solids) may be changed by varying the pH of the membrane-forming solutions. The effect of pH during dynamic membrane formation was investigated for Zr and PVP membranes. The optimum pH is 4.0 for Zr and 2.0 for PVP emulsions.

- The effect of emulsion temperature was investigated in the range of 25 to 65°C. In the investigated range of emulsion temperatures, the permeate flux increased with temperature during emulsion ultrafiltration through either Zr or PVP dynamic membranes. No appreciable changes in permeate quality and rejection with increase of temperature were observed.

- The effect of emulsion pH on ultrafiltration characteristics was investigated in the range of pH 5.5 to 9.5. There is a tendency to increased rejection of oil and solids by both Zr and PVP dynamic membranes during ultrafiltration of emulsion having pH reduced to about 5.5. This tendency is more pronounced for PVP membranes. At the same time, the average permeate flux decreased slightly when an emulsion of pH 5.5 was treated. Increase of emulsion pH to 9.5 did not result in measurable changes of ultrafiltration characteristics compared to those for emulsion in which pH was not adjusted (i.e., with pH about 6.5-7.5).

SUMMARY AND CONCLUSIONS (cont'd)

- Emulsion with different solids and oil concentrations were investigated in the LPRDM process using three types of dynamic membranes, Zr, PVP and PAA. The concentrations range of total oil was from about 800-850 mg/L to 7000-7500 mg/L, of total solids from about 1100-1600 mg/L to about 6450-7000 mg/L. Increased emulsion concentration affects the average permeate flux and quality, while no appreciable changes in rejection of either oil or solids were observed. The performance of PVP and PAA dynamic membranes (in terms of average permeate flux and rejection) is superior to that of Zr dynamic membrane.

- Steady-state operation of the LPRDM process can be carried out with bitumen/water/mineral emulsion, previously concentrated to about 90% of its initial volume reduction. Both PVP and Zr dynamic membranes can be used for treatment of the concentrated emulsion. The rejection of 85% to 96% oil and 55% to 72% solids can be achieved. The average permeate fluxes were as high as about 2.0 $m^3/m^2.d$ for Zr membrane and about 3.0 $m^3/m^2.d$ for PVP membrane, despite low transmembrane pressure of 10 psi (69 kPa).

- Complete regeneration of contaminated dynamic membrane can be achieved in-situ, without dismantling the unit.

The above regenerations allow not only multi-use of the same dynamic membrane at a permeability close to that of newly formed membrane, but also easy start-up and reuse of the LPRDM process after unscheduled shutdowns of the plant (part of which is the LPRDM process), which would be impossible otherwise without dismantling and recharging the unit with a new membrane.

It should be noted that for prefabricated membranes, regeneration of the contaminated membranes is a recognized problem. Even partial regeneration requires more complicated procedure. Quite often, the contaminated prefabricated membranes are replaced with new membranes and shutdown and dismantling of the unit or apparatus for a relatively long time is required, since a typical ultrafiltration or reverse osmosis industrial unit comprises numerous membrane modules.

CONCLUSIONS

Based on the results of experimental investigation obtained, the following conclusions and recommendations may be drawn:

- Effective treatment of bitumen/water/mineral emulsions associated with produced water in in-situ heavy oil recovery may be achieved using the LPRDM process.

- Recovery of water to reduction of initial emulsion volume by 90% and above can be obtained. The quality of permeate (i.e., separated water) and rejection of various components (e.g., oil and solids) are comparable with those obtained using prefabricated ultrafiltration membranes. The throughput (average permeate flux) is high, despite low transmembrane pressure drop.

- Bitumen/water/mineral emulsions in a wide range of oil and solids concentrations, pH and temperatures can be treated in the LPRDM process, with high permeate flux and rejection of both oil and solids.

- All three types of rotating dynamic membranes investigated, Zr, PVP and PAA can be used for bitumen/water/mineral emulsions treatment. PVP and PAA dynamic membranes, however, showed superior performance in comparison with Zr dynamic membrane.

- Complete regeneration of contaminated dynamic membranes can be carried out in-situ, without shutdown and dismantling the unit.

- The above in-situ regenerations allow not only multi-use of the same dynamic membrane at a permeability close to that of newly formed membrane, but also easy start-up and reuse of the LPRDM process after

CONCLUSIONS (cont'd)

unscheduled shutdowns of the in-situ heavy oil recovery plant (part of which would be the LPRDM process). Start-up would be impossible otherwise without dismantling the unit and forming a new membrane.

- Further investigation on application of LPRDM process for heavy oil/water/mineral emulsion is recommended based on positive results obtained in the course of the current exploratory study.

The next phase of investigation should, in particular, include:

- Treatment of heavy oil/water/mineral emulsions in the temperature range of 65° to 85°C (the typical temperature range of produced water during in-situ heavy oil recovery operations).

- Treatment of emulsions with a wide range of oil and solids concentrations. The range of heavy oil/water/mineral emulsion characteristics to be treated in the LPRDM process should be defined in collaboration with CANMET and major in-situ heavy oil producers in Canada.

- Long term operation of an LPRDM unit to determine the requirements for a commercial equipment prototype.

1.0 INTRODUCTION

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Canada possesses large deposits of heavy oil and oil sands and is fully engaged in the development of processes to economically exploit these major reserves (about 470 billion cubic metres as estimated in 1979 (1)). The most commonly used technique for in-situ recovery of heavy oil or bitumen (which is usually too viscous to flow at normal reservoir conditions) involves steam stimulation, either by intermittent steam injection (so-called "huff and puff" method) or by the combination of steam and fire flood method. The formation water and condensed steam which is produced with the oil, after phase separation, is called "produced water". The water-to-oil ratio may vary from 2:1 and up to 10:1. Therefore, the water requirements associated with in-situ heavy oil/bitumen requirements may be quite high (for example, total water requirements for the in-situ heavy oil recovery operation at Cold Lake, Alberta, are approximately 143 000 m^3/d (2)).

An economically efficient recycling of the produced water (including water treatment and reuse for steam generation) is a recognized problem in the industry. It is especially true for in-situ heavy oil recovery from some deposits in Western Canada located in short water supply areas (3).

After separation of free oil, the produced water usually contains emulsified oil, suspended solids, dissolved organic and inorganic substances. The oil/water/mineral emulsions* associated with in-situ production of heavy oil or bitumen are characterized by high stability. As shown in (4), the membranous skins formed by the clay and asphaltenes in such emulsions are responsible for

* Oil-in-water emulsions, i.e., water is the continuous phase.

high emulsion stability. The use of conventional de-emulsifying processes (electrostatic coalescence or surface active compounds, etc.,) proved to be ineffective.

Numerous methods and processes are being investigated for produced water treatment. Most of them have been adopted from classical boiler feedwater treatment principles. Among other processes being investigated is membrane technology. The recent investigation carried out by CANMET has shown the principal possibility to use prefabricated porous membranes for production of boiler-feed quality water from the wellhead bitumen/water/mineral emulsion containing about 4% bitumen (5). The subsequent extensive experimental work carried out by Zenon Environmental Inc., on a contract to CANMET, has further confirmed the possibility of produced water treatment by using several types of prefabricated porous membranes (6).

The low-pressure rotating dynamic membrane process is a variety of membrane technology that may be applied for separation of fine particles or substances of different molecular weight or specific gravity or both, from suspensions, emulsions, etc. Contrary to prefabricated porous membranes, the dynamic membranes are formed in-situ by a colloidal suspension or solution flowing (circulating) through a porous support and thus depositing a thin micron-size layer which acts as a filter membrane.

1.0 INTRODUCTION (cont'd)

The dynamic membranes and their properties were first investigated at Oak Ridge National Laboratory, U.S.A., and an extensive review on this subject was presented by Thomas (7).

Some of the advantageous features of dynamic porous membranes are as follows:

- Possibility to select an appropriate membrane-forming solution (suspension) and to adjust in-situ the porous structure, permeability and performance of the membranes by varying the formation conditions (e.g., pH, concentration) while using the same porous support.

- Possibility to accept wide range of pH and temperatures of the feed without reducing the performance characteristics, for most dynamic membranes.

- Selectivity in rejection of organic and inorganic substances, for some dynamic membranes.

- For rotating dynamic membranes, also possibility of regeneration or reconstitution in-situ of contaminated membrane, without shutdown or dismantling the unit or apparatus (see details in sub-sections 4.1.3 and 5.7).

1.0 INTRODUCTION (cont'd)

The low-pressure rotating dynamic membrane process (LPRDM) is a combination of an axial filter and a dynamic membrane formed and operated on a rotating porous surface of the filter.

The research and design work on an LPRDM unit and process was initiated by Dr. J.A. Mikhlin in 1977. Since that time, the mechanism of rotating dynamic membrane formation, as well as the performance of several types of these membranes, with both model compound and different industrial effluents, suspensions and waste waters, have been investigated in the LPRDM process. The effect of membrane formation conditions, physico-chemical properties of the treated feed and parameters in the LPRDM process performance have been studied (8,9). The methodology and procedure for regeneration or reconstitution of contaminated rotating dynamic membranes have been developed (10). Among the industrial effluents and suspensions investigated were black craft liquor from pulp and paper production, waste waters from food production (treated for reduction of BOD and COD to environmentally acceptable levels), recovery of chemicals (e.g., indigo blue dye from textile waste waters), ultrafiltration of electrodeposition paint in continuous car painting production lines and others (11,12).

The present work on exploratory investigation of the LPRDM process for treating bitumen/water/mineral emulsions associated with in-situ recovery of heavy oil/bitumen recovery was carried out by the Technical Development Department, SNC Inc., on a contract to CANMET.

2.0 OBJECTIVES OF THE STUDY

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The objectives of the study are as follows:

- to explore the feasibility of treating bitumen/water/mineral emulsion from a Canadian in-situ heavy oil/bitumen recovery operation using rotating dynamic membranes;

- to evaluate the effect of several factors on the LPRDM process performance during the treatment of the above emulsion;

- to compare the results obtained by Zenon Environmental Inc., using prefabricated ultrafiltration membranes (6) with those of the current SNC study.

For comparison purposes, the same sample of oil/water/mineral emulsion was used in both the Zenon Environmental Inc., and SNC investigations.

3.0 LOW PRESSURE ROTATING DYNAMIC MEMBRANE (LPRDM) PROCESS

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In this process, the feed (emulsion, suspension or solution) is filtered through a rotating dynamic porous membrane (previously formed on a rotating porous support) under the transmembrane pressure drop. As a porous support for dynamic membrane formation, either a porous film attached to the perforated surface of a rotating cylinder (rotor) or a rotor made from a porous material (e.g., porous metal, ceramics or plastics) are used. The filtrate (permeate) drains from inside the rotor (or rotors), while the reject remains in the outer space.

To separate the fine particles or substances of different molecular weight, the size of membrane pores has to be quite small and measures have to be taken to prevent fast clogging of membrane pores (for example, the smallest particles to be retained by ultrafiltration (UF) membranes are in the range 10 Å to 200 Å or 300 to 300 000 mol. wt based on globular protein; for reverse osmosis (RO) membranes, this range is 1 Å to 10 Å or below 300 mol wt (13)).

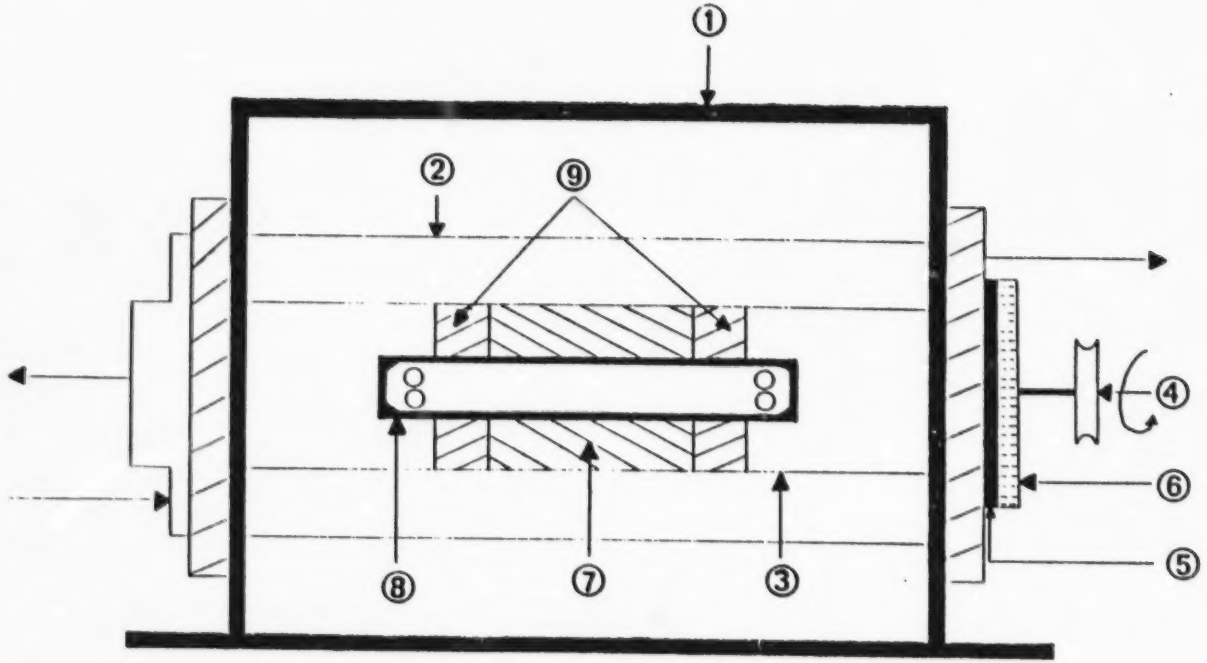
Cross-flow management (i.e., feed circulation along the membrane surface) is usually used in industrial UF or RO installations with prefabricated porous membranes to prevent the fast clogging of membrane pores and corresponding decline of permeate flux. The circulating (reject) steam has to be kept in a turbulent regime, thus requiring an excessive transmembrane pressure drop (above 100 psi or higher in UF installations, 400 to 1000 psi or higher in RO installations (13)).

3.0 LOW PRESSURE ROTATING DYNAMIC MEMBRANE (LPRDM) PROCESS (cont'd)

In the LPRDM process, the centrifugal forces developed in the vicinity of a rotating dynamic membrane are responsible for keeping the particles or higher molecular weight substances in the bulk of the reject stream and cross-flow management is not required to prevent the fast clogging of membrane pores. Therefore, a much lower transmembrane pressure drop (about 10-20 psi) could be applied to achieve the same or even higher permeate flux or throughput of the process.

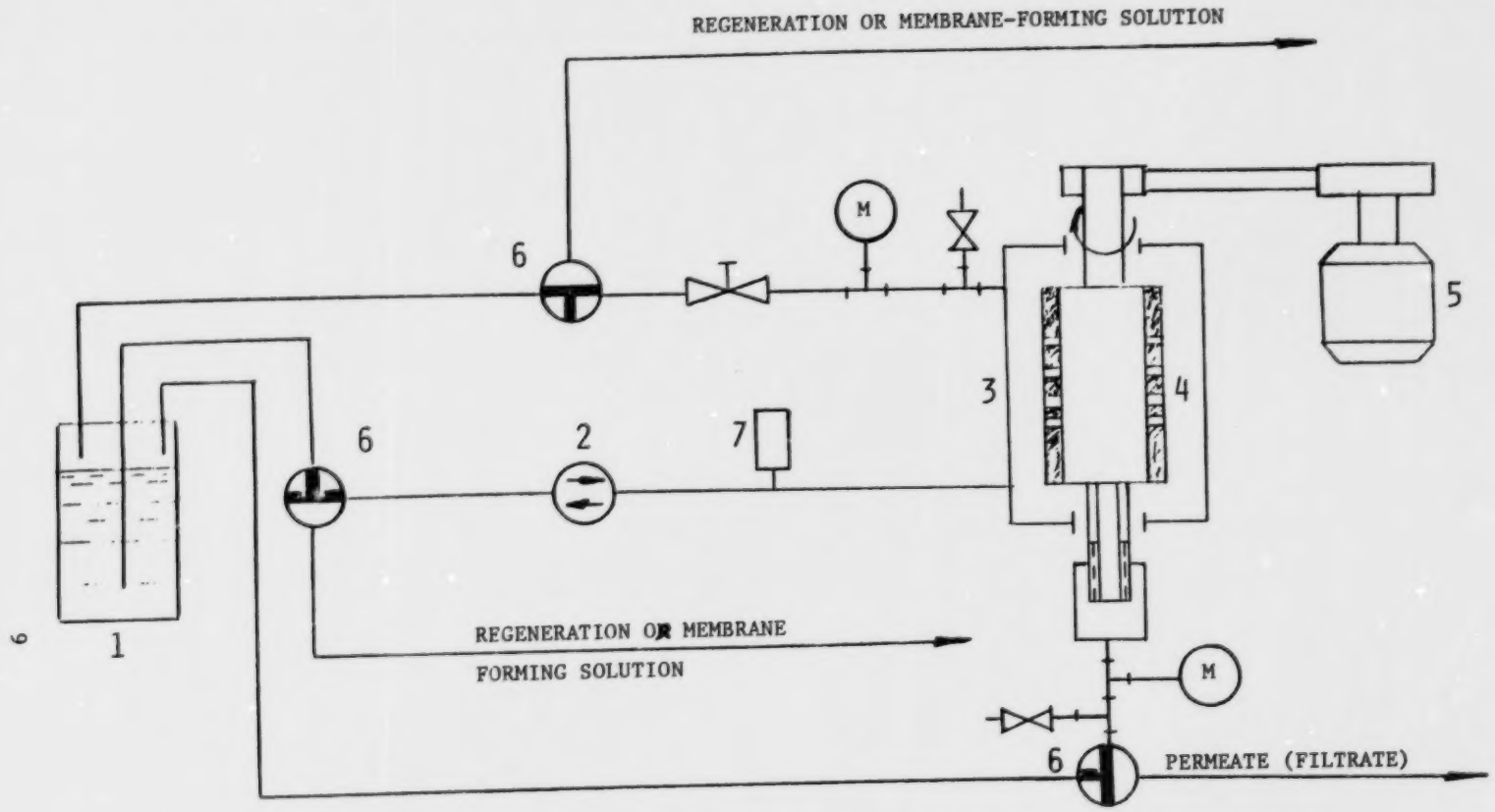
The schematic of the LPRDM unit used in the current study for exploratory investigation of heavy oil/water/mineral emulsion treatment is shown in Figure 1. The unit consisted of a rotating cylinder (5 cm OD, 20 cm long), over the central part of which 1300 holes (1 mm dia.) are drilled. This cylinder rotated within a concentric cylindrical housing. As a porous support for dynamic membrane formation a porous film (Acropor or Versapor, Gelman Instruments Inc.) was used. The film was wrapped around the central part of the rotor and held in place along its length by a clamp and taped at the end. The membrane filtration area was approximately 70 cm^2. To allow for the combined application of an electrical field and rotation of the membrane (i.e., centrifugal forces), if required, the rotating cylinder was fitted with bearings so that there was no electrical contact between the rotor and the housing.

The flowsheet of the LPRDM process as used in the current investigation is shown in Figure 2. The feed enters one end of the annulus between the rotor and the housing and leaves at the other. The filtrate drained from inside the rotor. The rotor could be rotated by a speed-controlled electrical drive at speeds up to



① SUPPORT FRAME
② MOTIONLESS HOUSING
③ ROTATING CYLINDER
④ DRIVE PULLEY
⑤ INSULATION
⑥ MERCURY CHAMBER
⑦ ACROPOR MEMBRANE
⑧ RETAINING CLAMP
⑨ TAPE

FIGURE 1 SCHEMATIC OF LOW-PRESSURE ROTATING DYNAMIC MEMBRANE UNIT



1 - FEED TANK
2 - REVERSIBLE PUMP
3 - LPRDM UNIT
4 - ROTOR
5 - ELECT. DRIVE
6 - THREE-WAY VALVE
7 - BUFFER

FIGURE 2 FLOWSHEET OF LOW-PRESSURE ROTATING DYNAMIC MEMBRANE PROCESS

4000 r/m. The LPRDM unit was fed by a peristaltic pump and a stabiliser (to reduce pulsations).

The transmembrane pressure was controlled manually with a valve on the reject (return) stream line. The emulsion feed vessel (6 litre volume) was immersed in a water bath kept at a controlled temperature (to ensure constant emulsion temperature during the tests). (The membrane-forming feed flask and a membrane regeneration solution flask are not shown in Figure 2.)

The following three modes of operation could be provided by this flowsheet (in either emulsion treatment or membrane formation/ regeneration cycles):

- closed loop (i.e., both the reject stream and filtrate returned to the feed vessel or flask);

- semi-closed loop (i.e., the reject stream returned to the feed vessel flask, while filtrate is removed);

- once-through operation (both reject stream and filtrate are removed but directed to different flasks).

The experimental and analytical procedures used are described in the following Section of the report.

4.0 PROCEDURES AND MATERIALS

4.0 PROCEDURES AND MATERIALS

The bitumen/water/mineral emulsion sample used in the experimental investigation was taken from the in-situ heavy oil recovery operation of Texaco Canada Resources, Fort McMurray, Alberta. As already noted, the sample was supplied to SNC by Zenon Environmental Inc., at the request of CANMET.

The initial emulsion sample characteristics are presented in Table 1. These analyses were performed by SNC at the start of the experimental program. The analytical procedures used are described in Section 4.3.

The characteristics of the emulsions used in various experimental tests in the LPRDM process are presented in respective sections of the report.

The quality objectives for boiler feedwater and other potential uses of produced water after treatment are presented in Table 2 (14).

4.1 Experimental Procedures

A brief description of experimental procedures used during dynamic membrane formation, emulsion treatment (ultrafiltration) and reconstitution/regeneration of contaminated membranes in the LPRDM process are presented below.

4.1.1 Dynamic Membrane Formation

Two types of dynamic membranes, zirconium hydrous oxide (further designated as "Zr") and poly-2 vinylpyridine (PVP) have been used in

Table 1

EMULSION CHARACTERISTICS

pH	7.5
TDO, mg/L	400
TSO, mg/L	400
TDS, mg/L	1015
TSS, mg/L	20
TOC, mg/L	400
SiO_2, mg/L	80
Ca, mg/L	0.64
Mg, mg/L	0.16
Fe, mg/L	120
Hardness, mg/L (calculated as $CaCO_3$)	2.2
Conductivity, uMhos/cm	720

Designations:

TDO - Total Dissolved Oil
TSO - Total Suspended Oil
TDS - Total Dissolved Solids
TSS - Total Suspended Solids
TOC - Total Organic Carbon

Table 2

QUALITY OBJECTIVES FOR BOILER FEEDWATER AND
OTHER POTENTIAL USES OF PRODUCED WATER

Parameter	Once-Through Steam Generator Note 4	Thermosludge Generator Note 4	Deep Well Disposal-Note 1	Irrigation	Cooling Water Make-up
Total Hardness, mg/L (calculated as $CaCO_3$)	< 0.5	100 - 2 000			2
Silica, mg/L	< 50	10 - 250			NOTE 2
Oil, mg/L	< 1	50 - 1 000	< 20	< 5	< 1
TDS, mg/L	8 000	1 000 - 50 000		< 1 000	
Suspended Solids, mg/L	0	10-500	< 5		NOTE 3
Dissolved O_2, mg/L	< 0.04	-			-
Iron, mg/L	< 0.05				

NOTES:

1. Water should be chemically compatible with formation waters
2. The product of Mg as $CaCO_3$ x SiO_2 < 35 000 mg/L
3. The product of Ca as $CaCO_3$ x SO_4 < 500 000 mg/L
3. Total suspended solids in the recirculating water 200 mg/L.
4. These are vendor recommendations. Some of these values have been exceeded in actual practice.

Source: "Review of Treatment and Recycling of Produced Water from Heavy Oil Fields", CH_2M Hill Canada Ltd., Report for U.S. D.O.E., and Energy, Mines and Resources Canada, 1981.

4.1.1 Dynamic Membrane Formation (cont'd)

most of the experiments. Also, in some tests, polyacrylic acid (PAA) dynamic membranes were formed.

During dynamic membrane formation, a membrane forming solution, adjusted to the desired pH, was circulated through the LPRDM unit (with new or regenerated porous film attached to the rotor) in a closed-loop mode of operation. The rotor speed and transmembrane pressure were kept constant, usually 2000 r/m and 10 psi (69 kPa) respectively. The membrane formation duration was about 30 minutes. After a dynamic membrane was formed, the remainder of the solution was pumped out of the LPRDM unit and back to the membrane solution flask. The unit was then rinsed with water.

The pH and initial concentrations of dynamic membrane-forming solutions used in most of the experiments were as follows:

- 0.2 wt% $ZrClO_2.8H_2O$ at pH 4, for Zr membranes;
- 100 ppm poly-2 vinylpyridine at pH 2, for PVP membranes;
- 100 ppm polyacrylic acid at pH 7, for PAA membranes.

In the experiments which were aimed to define the effect of pH or concentration of membrane-forming solution on the membrane performance during emulsion ultrafiltration, the pH or initial concentration of the solution are specified (if other than those listed above).

4.1.2 Emulsion Ultrafiltration

Emulsion ultrafiltration in the LPRDM unit was carried out after the dynamic membrane was formed as described in the previous section. Prior to this, a 5-litre sample of emulsion of desired concentration of oil and solids was prepared in the emulsion feed vessel, the pH and temperature of the emulsion were adjusted, if required, and then controlled during the ultrafiltration cycle at desired values. The transmembrane pressure and rotational speed in the unit were kept constant (usually 10 psi (69 kPa) and 2000 r/m respectively).

Samples of permeate (filtrate) and reject were taken concurrently for analyses; filtrate flow rates (permeate flux) were measured periodically (usually every 15 or 30 minutes).

Emulsion ultrafiltration in the LPRDM process was investigated in two modes of operation:

- semi-closed-loop, until the desired reduction of emulsion volume in the feed vessel was achieved;

- closed-loop during specified period of time, at constant concentration of the emulsion in the feed vessel.

Separation of produced water from the emulsion (emulsion ultrafiltration) was carried out to obtain the following three levels of initial emulsion volume reduction:

50%; 90%; above 90%

4.1.2 Emulsion Ultrafiltration (cont'd)

The emulsion sample supplied to SNC (see Table 1) was used for 50% volume reduction experiments. Five litres of this emulsion was placed every time in the feed vessel and ultrafiltration was carried out until 50% reduction of the volume was achieved.

As a feed for 90% volume reduction experiments, the emulsion remaining after completion of several 50% volume reduction tests was collected and placed in the emulsion feed vessel.

Similarly, for "above 90% volume reduction" experiments, the emulsion remaining after completion of several 90% volume reduction tests was collected.

In the closed-loop tests, the emulsion ultrafiltration was carried out continuously for a specified period of time (3½ and 8-9 hours).

The following range of process parameters were studied:

Emulsion temperature, ^{o}C		25 to 65
Emulsion pH		5.5 to 9.5
Emulsion concentration		
Total Oil	800-850	to 7000-7500 mg/L
Total Solids	1100-1600	to 6450-7000 mg/L

4.1.3 Dynamic Membrane Regenation

Regeneration in-situ of dynamic membranes was carried out in the LPRDM unit after the membranes were contaminated in the course of previous emulsion ultrafiltration through the membranes (usually corresponding to the reduction of initial emulsion volume of 90% or above).

The regenerating solution was pumped into the annulus between the rotor (with contaminated membrane) and the housing and circulated through the LPRDM unit at 10 psi transmembrane pressure and rotor speed of 2000 r/m. The completeness of regeneration was defined by achievement of steady-state permeate flux and then checked by comparison of emulsion ultrafiltration curves through the newly formed membrane and the same membrane after regeneration(s), using the same emulsion sample and ultrafiltration conditions.

Hot water (70^{o}-80^{o}C) was found suitable as the regenerating solution for all three types of dynamic membranes investigated. Alternatively, 1M NaOH solution for Zr membranes and 1M NaOH solution followed by 0.5% HCl solution were found suitable as regenerating solutions for Zr and PVP dynamic membranes respectively. The complete regeneration was usually achieved for a 15-20 minute cycle at the above specified conditions.

4.2 Analytical Procedures

The following analyses were carried out on the samples of emulsion (reject) and permeate flux (filtrate):

- Total Suspended Oil (TSO)
- Total Suspended Solids (TSS)
- Total Dissolved Oil (TDO)
- Total Dissolved Solids (TDS)
- Total Organic Carbon (TOC)
- Total Oil (TO)
- Total Solids (TS)
- Silicon
- Calcium
- Magnesium
- Iron
- Total hardness (calculated as $CaCO_3$)
- pH
- Conductivity

The analytical procedures used were as follows (15):

4.2.1 Total Suspended Oil (TSO) and Total Suspended Solids (TSS)

A known volume (V) of emulsion (reject) or permeate was filtered through a Whatman glass fibre filter. The latter was previously washed, ashed and weighed (W_1);

The filter was then dried at 90°C to a constant weight (W_2), and ashed at 450°C for 1 hour, and then again weighed (W_3). To volatilize oil without burning, the temperature in the furnace was first slowly increased up to 450°C.

4.2.1 Total Suspended Oil (TSO) and Total Suspended Solids (TSS) (cont'd)

The following expressions were used for calculations:

$$TSO = \frac{W_2 - W_3}{V} \qquad TSS = \frac{W_3 - W_1}{V}$$

4.2.2 Total Dissolved Oil (TDO) and Total Dissolved Solids (TDS)

After a crucible was ignited and weighed (W_1), all filtrate from the TSO and TSS determination (Volume V_1) was transferred into the crucible and dried at 60°C to constant weight. The crucible containing the dried sample was dessicated and again weighed (W_2).

Then, the dried sample was ashed for 1 hour at 450°C, the crucible containing the ashed sample was dessicated and weighed (W_3).

The TDO and TDS were calculated as:

$$TDS = \frac{W_3 - W_1}{V_1} \qquad TDO = \frac{W_2 - W_3}{V_1}$$

4.2.3 Total Oil (TO) and Total Solids (TS)

Total Oil is the sum of TSO and TDO;
Total Solids is the sum of TSS and TDS.

Note: In cases where TSO and TSS in the permeate flux were negligible, TO and TS in the permeate were also defined by omitting filtration through a Whatman glass fibre filter and drying and ashing the known volume of the sample (V_1) as described above for TDO and TDS determination.

4.2.4 Total Organic Carbon (TOC)

Analyses of total organic carbon were carried out using the TOC Analyzer, Dormann, Model DC-80, according to the procedure described in the operating manual of this analyzer.

4.2.5 Iron, Silicon, Calcium and Magnesium

The concentrations of these elements were measured using the Atomic Absorption Spectrophotometer, Perkin-Elmer, Model 403, following the procedures described in the operating manual.

4.2.6 Calculated Hardness (as $CaCO_3$)

Total hardness (H) was calculated based on the measured concentrations of calcium (Ca) and magnesium (Mg) to obtain equivalent calcium carbonate ($CaCO_3$), as follows:

$H = (2.5\ Ca - 4.116\ Mg)$ mg/L

4.2.7 pH and Conductivity

These measurements were made using the digital pH meter, Sargent-Welch, Model 600, and conductivity bridge, Yellow Springs Inc., (YSI), Model 31, following the procedures described in the corresponding operating manuals.

5.0 RESULTS AND DISCUSSIONS

5.1 Prescreening Tests

In the prescreening tests, samples of bitumen/water/mineral emulsion (see Table 1) were treated in the LPRDM process in the semi-closed mode of operation. Two types of dynamic membranes were used in the prescreening tests: Zr (0.2 wt% $ZrClO_2 \times 8\ H_2O$), membrane formation at pH 4 and PVP (100 ppm poly-2 vinylpyridine), membrane formation at pH 2).

Emulsion ultrafiltration in the prescreening tests was carried out to a reduction of initial emulsion volume to 50% and from 50% to 90%. In the dynamic membrane formation and emulsion ultrafiltration cycles, the transmembrane pressure was kept equal to 10 psi (69 kPa), and the speed of the rotor equal to 2000 r/m. The dynamic membrane formation and emulsion ultrafiltration procedures were described in Section 4.0.

Typical filtration curves are presented in Figure 3. The range of quality of the permeate (filtrate) and its average flux obtained during emulsion ultrafiltration prescreening tests with dynamic rotating membranes and those with prefabricated membranes obtained in (6) are presented in Table 3. The corresponding rejection of different components by the membranes are given in Table 4.

The rejections were calculated using the following expression:

$$R_{\%i} = \left(1 - \frac{C_{fi}}{C_{ri}}\right) \times 100$$

Here: $R_{\%i}$ - rejection of i-component, %

C_{fi} - concentration of i-component in filtrate (permeate) flux, mg/L;

C_{ri} - concentration of i-component in the reject stream, mg/L

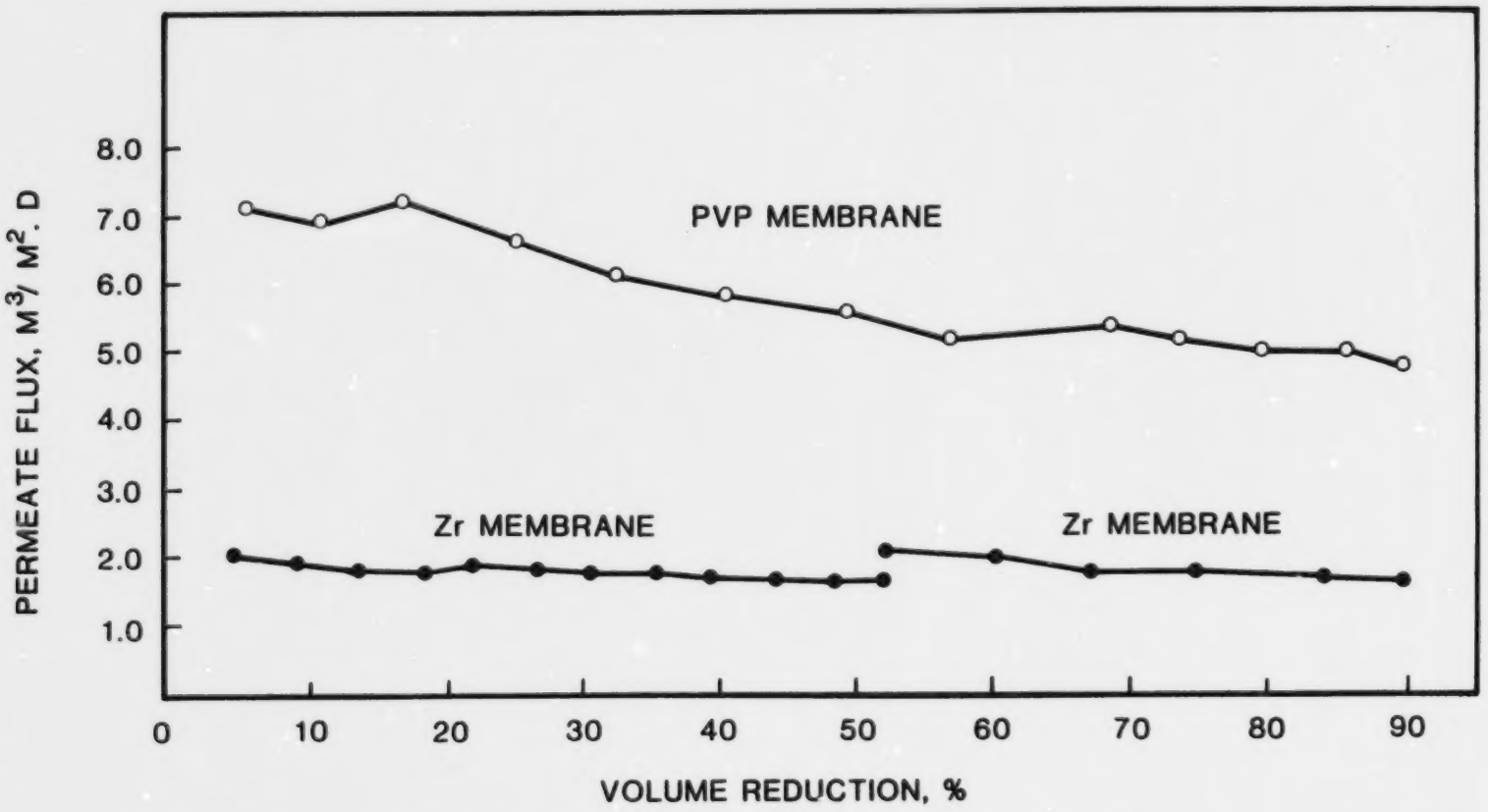


FIGURE 3 PERMEATE FLUX VS EMULSION VOLUME REDUCTION IN LOW-PRESSURE ROTATING DYNAMIC MEMBRANE PROCESS

Table 3

PERMEATE QUALITY AND AVERAGE FLUX DURING EMULSION ULTRAFILTRATION WITH PREFABRICATED AND DYNAMIC ROTATING MEMBRANES

No.	Membrane	TDS mg/L	TSS mg/L	TS mg/L	TDO mg/L	TSO mg/L	TO mg/L	TOC mg/L	SiO_2 mg/L	Hardness mg/L	Pressure Drop psi	Average Flux $m^3/m^2.d$
					DYNAMIC ROTATING MEMBRANES							
1	Zr	500-713	1-3	501-716	54-171	1-3	55-174	22-31	30-36	0.5	10	1.76
2	PVP	N/A	N/A	572-748	N/A	N/A	12- 84	24-63	29.2-60.0	2.8-3.4	10	3.60
					PREFABRICATED UF MEMBRANES							
3	Cellulose	605-845	N/A	N/A	N/A	N/A	N/A	42-140	40-69	0.6-28	100	0.28
4	CA	308-561	N/A	N/A	N/A	N/A	N/A	32-88	41-70	0.15-7.2	100	0.88
5	PS	399-475	N/A	N/A	N/A	N/A	N/A	32-66	36-70	0.15-8.7	100	0.88
6	CHP	362-470	N/A	N/A	N/A	N/A	N/A	21-76	39-68	0.15-4.5	100	0.45
7	PVDF	325-430	N/A	N/A	N/A	N/A	N/A	22-67	39-71	0.15-4.2	100	0.65
8	TFS	160-262	N/A	N/A	N/A	N/A	N/A	8-45	40-64	0.15-0.75	100	0.75

Notes: 1. Data on prefabricated UF membranes are taken from Zenon Env.Inc. report to CANMET Contract No. OSQ83-0023,Vol. II,March 31,1985

2. Feed emulsion pH = 5.5;Temperature 45^{o}-50^{o}C; Feed volume reduction up to 50%.

Table 4

REJECTIONS OF DIFFERENT COMPONENTS DURING EMULSION ULTRAFILTRATION WITH PREFABRICATED AND DYNAMIC ROTATING MEMBRANES

No.	Membrane	TDS	TSS	Rejection, % TS	TSO	TO	TOC	SiO_2	Hardness
				DYNAMIC ROTATING MEMBRANES					
1	Zr	45.6-58.6	95.6-96.7	35.8-55.2	99.6-99.9	86.9-88.3	87.4-92.3	60.5-73.5	78.3-94.5
2	PVP	N/A	N/A	56.7-58.9	N/A	82.5-86.2	74.5-94.2	78-84	73-83
				PREFABRICATED UF MEMBRANES					
3	Cellulose	19-44	N/A	N/A	N/A	N/A	91-96	19-44	23-86
4	CA	43-73	N/A	N/A	N/A	N/A	94-98	18-39	67-99
5	PS	51-64	N/A	N/A	N/A	N/A	95-98	18-42	82-99
6	CHP	52-68	N/A	N/A	N/A	N/A	94-98	20-42	81-99
7	PVDF	59-70	N/A	N/A	N/A	N/A	96-98	16-44	84-99
8	TFC	74-86	N/A	N/A	N/A	N/A	97-98	18-42	98-99

Notes: 1. Data on prefabricated UF membranes are taken from Zenon Env.Inc. report to CANMET Contract No. OSQ83 - 0023,Vol. II, March 31,1985

2. Feed emulsion pH = 5.5; Temperature 45^{o}-55^{o}C; Feed volume reduction up to 50%.

5.1 Prescreening Tests (cont'd)

The samples of reject and permeate were taken concurrently, as described in Section 4.0.

Examination of the results obtained during prescreening tests on treatment of designated bitumen/water/mineral emulsion in the LPRDM process have shown the following:

a) Effective treatment of such emulsions, with reduction of initial emulsion volume as high as 90% and above may be achieved with both Zr and PVP rotating dynamic membranes.

b) The rotating dynamic membranes throughput (permeate flux) is high despite low transmembrane pressure. The quality of permeate flux and rejection of various components are comparable with those obtained using prefabricated ultrafiltration membranes for the same emulsion treatment.

c) The average permeate flux at the LPRDM unit is 4- to 8-fold higher than that obtained with prefabricated ultrafiltration membranes, despite 10-fold lower transmembrane pressure (10 psi in the LPRDM unit versus 100 psi with prefabricated UF membranes).

5.2 Effect of pH of Membrane-Forming Solutions

The effect of pH of membrane-forming solutions during formation of Zr and PVP rotating dynamic membranes in the LPRDM unit on performance of the membranes is shown in Table 5.

Table 5

EFFECT OF pH OF MEMBRANE
FORMING SOLUTION (SUSPENSION)

No.	Dynamic Membrane		Emulsion Ultrafiltration		
	Type	Formation pH	Average Flux $m^3/m^2.d$	Rejection, %	
				Oil	Solids
1	Zr	4.5	2.8	81	31
2		4.0	1.9	86	55
3		4.0	2.0	90	66
4	PVP	4.0	2.4	63	61
5		4.0	2.1	81	47
6		2.0	3.6	97	59
7		2.0	4.1	97	61

Note: Emulsion volume reduction 50%, Temperature 50°C, pH 5.5

Hereinafter, rejection of TSO plus TDO (or of TO, if total suspended oil in permeate was negligible) is denoted as oil rejection; rejection of TSS plus TDS (or of TS, if total suspended solids in permeate was negligible) is denoted as solids rejection. The transmembrane pressure of 10 psi (69 kPa) and rotational speed of 2000 r/m maintained during both dynamic membrane formation and emulsion ultrafiltration cycles.

5.2 Effect of pH of Membrane Forming Solutions (cont'd)

It may be seen that for the Zr rotating dynamic membrane, increase pH above the conventional value of 4.0 resulted in higher permeate flux but reduced rejections; for the PVP rotating dynamic membrane; on the contrary, deviation of pH of membrane-forming solution up from the conventional value of 2.0, decreased the permeate flux. Also, quality of the product was lower.

5.3 Effect of Emulsion Temperature

The effect of emulsion temperature was investigated in the range of 25° to 55°C. The results are summarized in Table 6. Some tests were also carried out at an emulsion temperature of 65°C.

Table 6

EFFECT OF EMULSION TEMPERATURE

No.	Dynamic Membrane		Emulsion Ultrafiltration				
	Type	Formation pH	pH	Temp. °C	Average Flux $m^3/m^2.d$	Rejection, %	
						Oil	Solids
1	Zr	4.0	6.8	25	1.1	92.1	68.7
2	Zr	4.0	6.8	55	2.1	88.2	69.4
3	PVP	2.0	6.8	25	3.4	94.2	56.0
4	PVP	2.0	6.8	55	4.3	93.3	54.4

Note: Emulsion volume reduction 50%.

5.3 Effect of Emulsion Temperature (cont'd)

In the investigated range of emulsion temperatures, the permeate flux increased with temperature during emulsion ultrafiltration through either Zr or PVP dynamic membranes. No appreciable changes in permeate quality and rejection with increase of temperature were observed.

5.4 Effect of Emulsion pH

The effect of emulsion pH on ultrafiltration characteristics was investigated in the range of pH 5.5 to 9.5.

Prior to these experiments, emulsion stability tests in the above range of pH were carried out by adjusting the samples of emulsion (as delivered, and reduced to 50% and 90% of initial volume) to pH 5.5; 7.0; 8.0; 9.5 and leaving the samples at room temperature for 300 hours. For some emulsion stability tests, the samples were heated to about 70°C, adjusted to pH 5.5 and kept at this temperature and pH for 8 hours. No signs of emulsion destabilization were observed at the above described conditions.

The effect of emulsion pH between 5.5 and 7.8 on emulsion ultrafiltration (average flux and rejection of oil and solids) through Zr and PVP dynamic membranes is shown in Table 7.

Table 7

EFFECT OF EMULSION pH

No.	Dynamic Membrane	Emulsion Ultrafiltration			
		pH	Average Flux $m^3/m^2.d$	Rejection, %	
				Oil	Solids
1	Zr	5.5	1.9	86	55
2	Zr	5.5	2.0	90	66
3	Zr	6.8	2.1	88	69
4	Zr	7.0	2.0	86	50
5	PVP	5.5	3.6	97	59
6	PVP	5.5	4.1	97	61
7	PVP	6.8	4.3	93	54
8	PVP	7.8	7.0	78	50

Notes: pH of membrane-forming solutions: 4.0 for Zr and 2.0 for PVP dynamic membranes; emulsion temperature 50°C; emulsion volume reduction 50%.

It may be seen that there is a tendency to increased rejection of oil and solids by both Zr and PVP dynamic membranes during ultrafiltration of emulsion having pH reduced to about 5.5. This tendency is more pronounced for PVP membranes. At the same time, the average permeate flux decreased slightly when an emulsion of pH 5.5 was treated. (Test 8 should likely be considered as anomalous.)

5.4 Effect of Emulsion pH (cont'd)

Increase of emulsion pH to 9.5 did not result in measurable changes of ultrafiltration characteristics compared to those for emulsion in which pH was not adjusted (i.e., with pH about 6.5-7.5).

5.5 Effect of Emulsion Concentration

Emulsion of different levels of concentration with solids and oil was investigated in the LPRDM process using three types of dynamic membranes, Zr, PVP and PAA. The concentrations of total oil was changed from about 800-850 mg/L to 7000-7500 mg/L, of total solids from about 1100-1600 mg/L to about 6450-7000 mg/L. The increased concentrations of emulsion were achieved by ultrafiltration of emulsion as supplied to SNC to reduction of its initial volume to 50%, 90% and above, using the procedures described in sub-section 4.1.2.

Examination on the data presented in Table 8 shows that:

- Bitumen/water/mineral emulsions with a relatively wide range of oil and solids concentration may be treated in the LPRDM process. High permeate flux and rejection of both oil and solids may be achieved.

- Increased emulsion concentration affects the average permeate flux and quality, while no appreciable changes in rejection of either oil or solids were observed.

- The performance of PVP and PAA dynamic membranes (in terms of average permeate flux and rejection) is superior to that of Zr dynamic membrane.

Table 8

EFFECT OF EMULSION CONCENTRATION

No.	Dynamic Membrane		Feed Concentration			Emulsion Ultrafiltration				Average Flux
	Type	Formation pH	Volume Reduction %	Oil mg/L	Solids mg/L	Permeate		Rejection, %		
						Oil mg/L	Solids mg/L	Oil	Solids	$m^3/m^2.d$
1	Zr	4.0	50	870	1610	114	716	86	55	1.9
2		4.0	90	2275	2448	216	924	91	62	1.6
3		4.0	> 90	3240	6470	150	2835	95.4	56.2	1.7
4	PVP	2.0	50	480	1820	12	748	97.5	59	3.6
5		2.0	90	1256	2680	40	828	96.8	60	2.5
6		2.0	> 90	1858	3012	200	850	89.2	72	2.3
7	PAA	7.0	> 90	3095	3065	210	750	93.2	75.5	5.1
8		7.0	> 90	7210	3255	135	1025	98.1	68.5	3.8

NOTE: Process conditions: Pressure drop 10 psi (69 kPa), rotational speed 2000 r/m, emulsion pH 5.5 and temperature 50^{o}-55^{o}C.

5.6 Closed-Loop Tests

To define operability of the LPRDM process with emulsion concentrated by 90% reduction of its initial volume, several continuous runs of such emulsion samples have been carried out using both Zr and PVP dynamic membranes.

Figure 4 shows filtration curves (permeate flux vs time) for two 8.5-hour runs.

In both runs, the operating conditions were: emulsion temperature 50°C; pH 6.5-6.9; oil concentrations (total oil) about 1850 mg/L and 2300 mg/L for Zr and PVP membranes respectively. Total solids concentrations were about 2200 mg/L for the test with Zr membrane and about 5700 mg/L with PVP membrane.

The rejections obtained were: 93% to 96% of oil and 62% to 72% of solids for Zr dynamic membrane; 85% to 88% of oil and 55% to 59% of solids for PVP dynamic membrane.

The tests confirmed that steady-state operation of the LPRDM process can be carried out with bitumen/water/mineral emulsion, previously concentrated to about 90% of its initial volume reduction. Both PVP and Zr dynamic membranes can be used for treatment of the concentrated emulsion. The rejection of 85% to 96% oil and 55% to 72% solids can be achieved. The average permeate fluxes were as high as about 2.0 $m^3/m^2.d$ for Zr membrane and about 3.0 $m^3/m^2.d$ for PVP membrane, despite low transmembrane pressure of 10 psi (69 kPa).

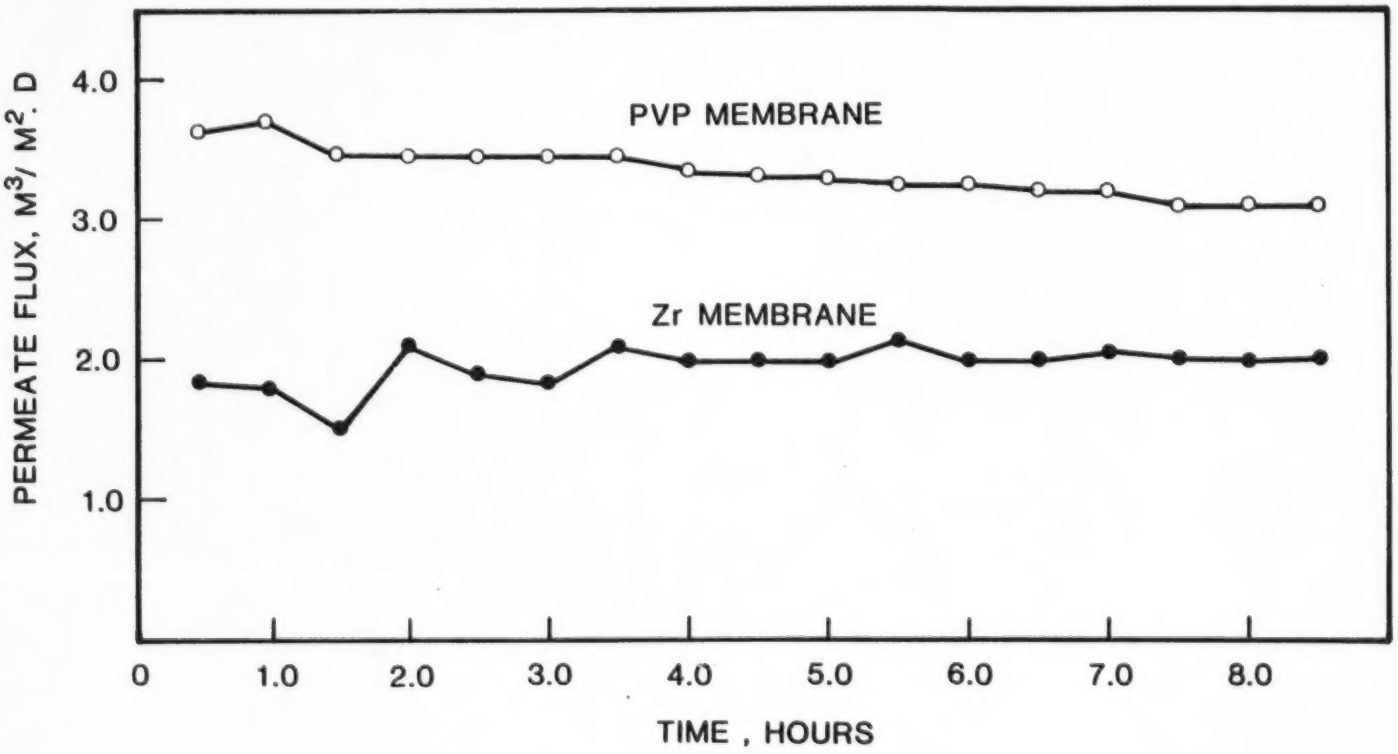


FIGURE 4 EMULSION ULTRAFILTRATION IN LOW-PRESSURE ROTATING DYNAMIC MEMBRANE PROCESS (CLOSED-LOOP AT 90% EMULSION VOLUME REDUCTION)

5.7 Dynamic Membrane In-Situ Regeneration

The procedures for in-situ regenerations of contaminated dynamic membranes after emulsion ultrafiltration in the LPRDM process have been described in Sub-section 4.1.3, Figure 5 presents filtration curves obtained during seven consecutive UF cycles, first through newly formed PVP dynamic membrane (run No. 1) and then after regeneration in-situ of this membrane (runs No. 2 to 7). During each UF cycle, the emulsion was concentrated from 50% to 90% of its initial volume.

The rejections obtained were: in the range of 90% to 97% for total oil; and between 60% and 69% for total solids. No appreciable influence of membrane regeneration on degree of rejection was observed.

1M NaOH aqueous solution followed by 0.5% HCl solution were used for membrane regeneration in these tests.

It may be seen, that complete regeneration of contaminated dynamic membrane can be achieved in-situ, without dismantling the unit.

Similar regeneration in-situ results were obtained with contaminated Zr dynamic membranes using procedures described in Sub-section 4.1.3.

The above simple and quick in-situ regenerations allow not only multi-use of the same dynamic membrane at a permeability close to that of newly formed membrane, but also permit easy start-up and reuse of the LPRDM process after unscheduled shut-downs of the plant (part of which is the LPRDM process), which would be impossible otherwise without dismantling and recharging the unit with a new membrane.

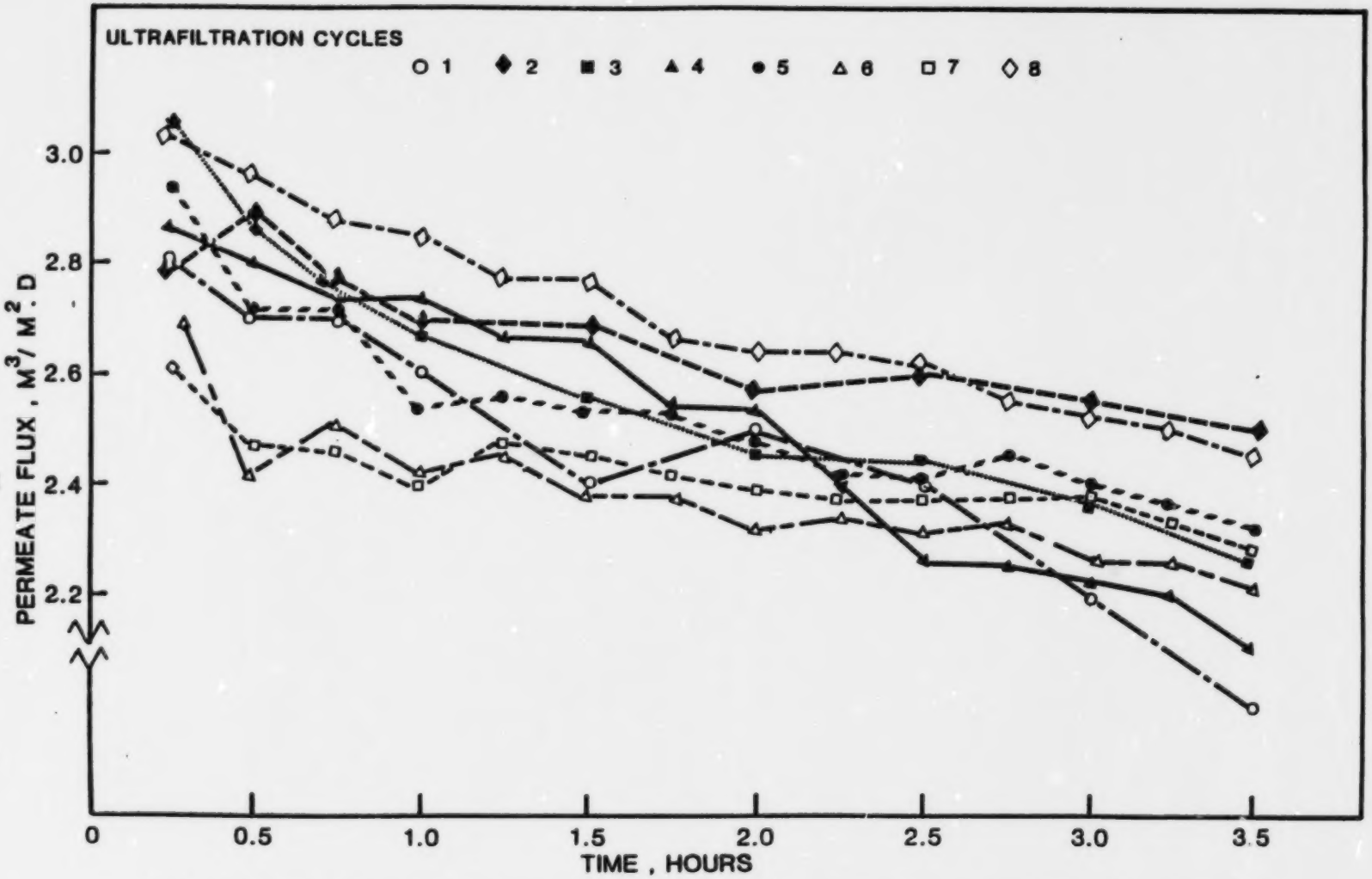


FIGURE 5 EMULSION ULTRAFILTRATION IN LOW- PRESSURE ROTATING DYNAMIC MEMBRANE PROCESS THROUGH NEWLY FORMED AND REGENERATED PVP DYNAMIC MEMBRANE

5.7 Dynamic Membrane In-Situ Regeneration (cont'd)

It should be noted that for prefabricated membranes, regeneration of the contaminated membranes is a recognized problem. Even partial regeneration requires more complicated procedure. Quite often, the contaminated prefabricated membranes are replaced with new membranes and shutdown and dismantling of the unit or apparatus for a relatively long time is required, since a typical ultrafiltration or reverse osmosis industrial unit comprises numerous membrane modules.

5.8 Power Consumption

The power consumed in the LPRDM unit is used to overcome the mechanical friction in the bearings and seals, as well as the shear forces at the surface of the rotor. Therefore, the total power consumption P may be expressed as a sum of two components designated as P_M (let us call it "Mechanical Power") and P_sL (let us call it "Shear Power per unit of length of the rotor" multiplied by the length of the rotor):

$$P = P_M + P_sL \qquad (2)$$

where P_M is the mechanical power which depends on rotational speed, type and maintenance of the bearings and seals, pressure of the fluid and diameter of the rotor, but independent of length of the rotor and fluid viscosity

P_s is the shear power per unit of length of the rotor and depends on the rotational speed, diameter of the rotor, density and viscosity of the fluid, but independent of the pressure

L is the length of the rotor.

5.8 Power Consumption (cont'd)

The power required to rotate a cylinder in air is a very small percentage of that required to rotate the same cylinder in water (or emulsion), because the density and viscosity of air are respectively, 1000-fold and 50-fold lower than that for water. Therefore, it may be assumed that when the annular space between the rotor and outside cylinder in LPRDM unit is filled with air at pressure used in the experiments, the power consumption measured will represent the "mechanical power" P_M only, since the "shear power" in this case will be very small as compared to filling the space with water or more viscous liquid. Under these assumptions, the difference between power consumptions measured with liquid and air at the same operating conditions (rotational speed and pressure) will represent the "shear power" $P_s L$.

Determination of power consumption for rotating of filter element in LPRDM system was carried out using LPRDM unit with mechanical torque meter, type TDS-DN-TM1 (General Thermodynamics Corporation, Plymouth, Mass.), coupled directly between the shafts of the electrical drive and the rotating filter element (rotor) of the unit. During P_M evaluation, air was used as the fluid in the annular space between housing and rotor; water was used during evaluation of "shear power per unit of length of the rotor" $P_s L$ (it was assumed that the viscosity of the emulsion used at the process conditions investigated is close to that of water).

5.8 Power Consumption (cont'd)

Power consumption was calculated from direct readings of torque meter (in OZ-in.) using the formula:

$$P = \frac{M \times N \times 745.7}{16 \times 62025} \qquad (1)$$

where: P = power consumption, Watts,
M = torque, oz-in.,
N = rotational speed, rpm,
745.7 = conversion from hp to Watts,
16 = conversion from oz-in. to lb-in.

The power consumptions measured at 69 kpa pressure and 2000 rpm speed were as follows:

P_M = 15 Watts

P_sL = 13.5 Watts

A change of water temperature in the range of 25-65°C did not result in a measurable variation of P_sL.

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